

RECYCLING BISULFITE BRINES USED IN SWEET CHERRY PROCESSING

ABSTRACT

Recycling was evaluated as a means of alleviating pollution from cherry processing brines. Brines were reclaimed by filtration, treatment with activated carbon, and addition of SO_2 and lime. Cherries processed with reclaimed brine were similar in composition, color, and firmness to controls. The capacity of the reclamation system was estimated. Reclaimed brine became discolored if iron exceeded 30–40 ppm. SO_2 losses were higher in full strength brines than in weaker brines. Brined cherries repacked in water lost more SO_2 than did brined controls and became discolored during storage. Maraschino cherries prepared from water-packed cherries were less firm than controls. Design and engineering studies based on these data are in progress.

INTRODUCTION

STRONG calcium bisulfite brines are used by the sweet cherry processing industry to bleach, firm and preserve fruit which will be made into maraschino, candied, and glacé cherries. Brines may be prepared from gaseous SO_2 or NaHSO_3 and typically contain 15,000 ppm SO_2 at pH 2.8 (Watters, 1975; Tennes et al., 1975). The disposal of spent brine, which may still contain several thousand ppm SO_2 , represents a serious problem for the cherry processing industry. Such processing wastes are corrosive, have a high chemical oxygen demand, and develop objectionable odors under anaerobic conditions due to the growth of sulfur-reducing bacteria (Soderquist, 1971). In Michigan the brine disposal problem is compounded by the close proximity of many cherry processors to residential and resort areas and by the lack of adequate municipal sewage treatment facilities.

One possible solution to the disposal problem, reclamation of spent brine by activated carbon treatment, was investigated at Oregon State University (Beavers et al., 1970; Soderquist, 1971). Research was initiated by the USDA and by Michigan State University to determine the feasibility of reclamation and other approaches to the brine disposal problem in Michigan. The specific objectives of the research reported herein were to obtain quantitative measurements of the performance and capacity of the brine reclamation system, and to determine the impact of other factors affecting the feasibility of the system including iron contamination, brine storage stability, and the practice of water packing brined cherries.

MATERIALS & METHODS

Preliminary experiments

A used cherry processing brine, which originated in Michigan during the summer of 1973, was obtained for preliminary studies. This brine had been prepared from a prepackaged dry mix (Tennes et al., 1975) and had been used to bleach dark-skinned varieties of cherries.

The brine was reclaimed by passage through a 75 × 5.1 cm glass column containing 700g activated carbon (Calgon 300; Merck & Co., Inc.). The reclaimed brine was brought to full strength by the addition of SO_2 and $\text{Ca}(\text{OH})_2$ to compensate for losses due to dilution, evaporation, and chemical reaction. The control, new dry mix brine, was prepared using a commercial formulation: add 24.4g NaHSO_3 , 3.2g fu-

maric acid, and 20.8g CaCl_2 (anhydrous), in the order listed, to one liter of water.

Cherry brining experiments were carried out in Pennsylvania and Michigan orchards during the summer of 1974. Schmidt, Windsor, Bing and Napoleon cultivars, grown near Traverse City, Mich., were stemmed and immersed in reclaimed and control brines using approximately 1 kg fruit per liter brine. Similar tests were conducted in eastern Pennsylvania using Windsor, Oxheart, Napoleon, and Emperor Francis cherries. The brined cherries were stored at room temperature until the winter of 1974–75 when they were finished into maraschinos and evaluated.

System capacity and performance

During the Fall of 1974, a larger scale brine reclamation system was assembled for the quantitative measurement of system capacity and performance (schematic diagram on Fig. 1). The system consisted of a glass column (7 × 75 cm), containing 60 cm of 0.45–0.55 mm water filtration grade sand, in series with three glass columns, each 6.4 × 115 cm, containing acid-washed activated carbon (Pittsburgh CPG Granular Carbon, 12 × 40 mesh, Calgon Corp., Pittsburgh, PA). The total weight of activated carbon was 3938g. The columns were plugged with glass wool at both ends and were connected by tygon tubing attached to ground glass ball joints. Brine was pumped through the system (from the bottom of each column to the top of the succeeding column) with a MasterFlex Peristaltic Pump (Model 7015) at a flow rate of approximately 40 ml/min, controlled by a stopcock at the bottom of the last column.

Dry mix brines, which had been used to brine Windsor and Napoleon cherries (1974 Michigan pack), were passed through the sand/activated carbon system. The daily and cumulative volume of treated brine was recorded, and samples were taken from each column at the beginning and completion of each day's operation to be analyzed for pH, SO_2 , soluble solids, and transmittance. All brine treated by this system was retained; portions of brine from the same source and having similar composition after treatment were pooled. Following treatment or immediately prior to the next cherry brining season, the reclaimed brines were adjusted to full strength as described previously. Brines were stored at room temperature and were analyzed periodically to determine their stability.

Field tests were conducted during June of 1975 to evaluate the performance of the reclaimed brines. Windsor, Schmidt and Napoleon cherries were obtained at local orchards and immediately brined using 1 kg stemmed fruit/liter brine. The brined cherries were held at room temperature until the winter when they were processed into maraschinos and evaluated.

Maraschino process

The following process was used to prepare maraschino cherries: (1) Drain, pit, and weigh 100g brined cherries. (2) Soak cherries in three successive portions of water (cherries/water = 45/55 by weight) for approximately 18 hr per soak at room temperature. (3) Add a 60.6 ml portion of 40 brix sirup prepared by combining 470g sucrose, 4.29g sodium benzoate, 0.281g FD&C Red No. 4, 706g H_2O , and sufficient citric acid to adjust the pH to 3.5. Equilibrate cherries in sirup for 2 days. (4) Add 0.00284g FD&C Red No. 40. (5) Add three successive 25.3g portions of 74° Brix sucrose solution (warmed to dissolve) on each of 3 days. (6) Adjust the sugar concentration in the product to 40° Brix by a final addition of 74° Brix sirup and adjust the pH to 3.5 with citric acid as required. (8) Pasteurize by heating to 65.5°C.

Water-packed brined cherries

Windsor and Napoleon cherries, packed in SO_2 and dry mix brines during the 1973 season in Michigan, were obtained from a commercial

Table 1—Evaluation of brined Michigan cherries in 1974 experiments

Cherry variety	Brine	Brine composition after equilibration				Shear press ^a		Reflectance ^c		
		pH	SO ₂ (ppm)	Ca (ppm)	Soluble solids (%)	Pitting force ^b	Firmness ^c	Rd	a	b
Schmidt	Control	3.1	3450	3120	10.5	14.2	9.2	9.0	57.9	22.0
	Reclaimed	3.0	4190	4040	12.8	14.7	8.9	8.8	56.4	21.8
Windsor	Control	3.4	3550	3740	11.1	10.4	8.9	7.8	53.4	21.4
	Reclaimed	3.4	3400	4200	13.7	10.2	7.7	7.8	53.8	21.8
Bing	Control	3.5	4140	4140	10.1	10.5	11.1	7.3	48.4	20.1
	Reclaimed	3.4	4530	4740	12.9	11.5	10.0	7.4	49.0	20.2
Napoleon	Control	3.1	3300	2860	10.5	14.4	8.7	8.8	58.0	22.2
	Reclaimed	2.9	3390	3720	13.0	15.7	10.2	8.6	55.6	21.9

^a Area under curve / g cherries^b Brined cherries^c Maraschino cherries

briner in November 1974. Samples were drained and repacked in distilled water using equal weights of cherries and water in 8 and 32 oz jars. The water packed samples and brine-packed controls were periodically evaluated for brine composition and appearance. At the completion of the storage period, the cherries were finished using the previously described maraschino process, and the final products were evaluated for firmness and color.

Analytical methods

Sulfur dioxide was determined by iodimetric titration as described by Payne et al. (1969). Calcium in brines prepared in preliminary experiments (1974) was determined by the EDTA titrimetric procedure of Payne et al. (1969). In subsequent studies, calcium was determined by the lanthanum chloride atomic absorption procedure described by Perkin-Elmer (1973) using a Perkin-Elmer Model 306 atomic absorption spectrophotometer. Iron also was determined by atomic absorption using the method of addition described by Perkin-Elmer to compensate for brine sodium. Soluble solids in brine were measured with a Bausch & Lomb Abbe-3L refractometer.

All spectrophotometric measurements on brine samples were performed with a Bausch & Lomb Spectronic 88 spectrophotometer using distilled water as the blank. Percent transmittance at 335 nm, the wavelength of minimum transmittance used by Beavers et al. (1970) as an index of quality for reclaimed brine, was determined on brine samples without pH adjustment. The absorbance at 510 nm of brines adjusted to pH 1.00 with concentrated H₂SO₄ was used as an index of anthocyanin concentration. To avoid interference from CaSO₄ precipitation, spectrophotometric measurements were performed rapidly. In addition, the absorbance at 540 nm of brine adjusted to pH 8.00 was used to evaluate reclaimed brine after it was observed that some samples which were colorless at pH 1.0 became intensely blue at the higher pH. An absorption maximum at 540 nm was observed in these samples. The brine samples were adjusted to pH 8.0 with Ca(OH)₂, filtered through Whatman 2v paper to remove precipitated CaSO₄, and readjusted to pH 8.00 with several drops of 1N H₂SO₄. A linear relationship was found between absorbance at 510 and 540 nm and brine concentration in samples of Windsor brine diluted fivefold to 100-fold and adjusted to pH 1.00 and 8.00, respectively. Similar results were obtained at 540 nm with recycled Windsor brine, diluted as much as fivefold and adjusted to pH 8.00.

Reflectance measurements were made on maraschino cherry homogenates using a Gardner Automatic Color Difference Meter (Model-AC2A 200) standardized with a red tile (SCR0020) for which Rd, a, and b measurements were 7.0, 59.7 and 19.3, respectively. The homogenates were prepared by blending 150g drained fruit for 1 min at high speed with a Waring Blender.

Pitting force and firmness measurements were made with an Allo-Kramer Shear Press (Model S-2HE), an Allo E-2EZ amplifier, and an Esterline Angus E 1101E recorder fitted with a 247-R Disc Integrator, originally designed for potato texture research as described by Ross and Porter (1969). Cherries were placed (sutures facing up) in 10 cavities in a specially designed aluminum cell and were pitted with stainless steel punches, simulating the action of commercial pitting machines. The

firmness of pitted maraschino cherries was determined on fruit oriented in the cell cavities so that the punch stroke would be perpendicular to the pitting hole. A ram speed of 0.12 in./sec, and a 100 lb proving ring were used. Results were expressed as area under the shear-force curve (counts) per gram of sample.

RESULTS & DISCUSSION

Preliminary experiments

Preliminary experiments carried out in 1974 demonstrated that used brines could be reclaimed by activated carbon treatment without appreciably altering their composition or their performance (Table 1). Following equilibration with cherries, the reclaimed brine tended to contain higher concentrations of SO₂, calcium, and soluble solids than did the control brines. Cherries brined in reclaimed and control brines were similar with respect to pitting force, as measured with the modified shear press. Maraschino cherries prepared from the brined samples were similar in firmness and in color; differences were related to cherry variety rather than to the brine. Results obtained with cherries from Michigan and Pennsylvania were similar. Beavers et al. (1970) reported that brined cherries processed with reclaimed brine were more firm and similar in color to cherries processed with new brine; the soluble solids content of the reclaimed brine was higher than that of the new brine after equilibration with the brined fruit.

System capacity and performance

The capacity of the three column activated carbon system was evaluated by treating large volumes of used brine and measuring changes in the composition and performance of the treated brine. The first brine to be treated, a highly pigmented brine which had contained Windsor cherries, was reclaimed with no change in pH, SO₂, or soluble solids after 282 liters had been treated. The pigment concentration in the brine was greatly reduced by the three columns and did not increase during the treatment (Table 2). However, the concentration of components absorbing at 335 nm in the effluent from the first column showed a progressive increase as the activated carbon became saturated (Fig. 2). A brine which had been used to process less highly pigmented Napoleon cherries was reclaimed after the treatment of the Windsor brine was completed. As the volume of Napoleon brine through the system increased, a large change in transmittance at 335 nm and absorbance at 540 nm (at pH 8) occurred (Fig. 3). These indications of increasing brine pigmentation paralleled the complete saturation of the second column (at 600 liters) and the gradual failure of the third column. The experiment was terminated at 641

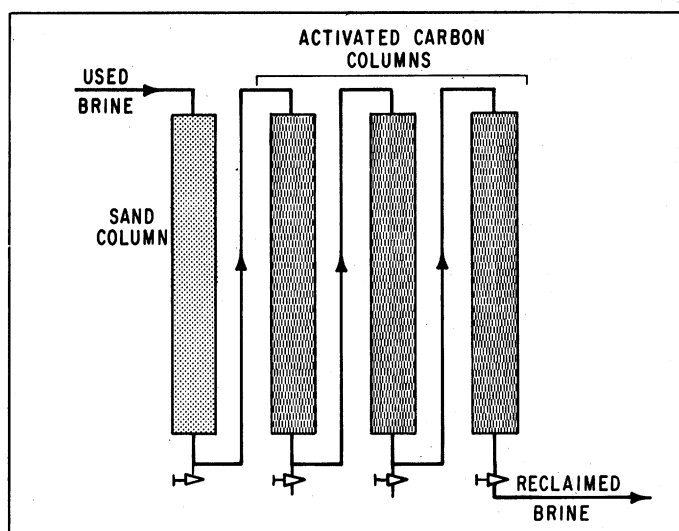


Fig. 1—Schematic diagram of brine reclamation system.

liters, and the reclaimed brine portions obtained during each day's operation were pooled into five fractions on the basis of the spectrophotometric measurements (Table 2) and retained for further evaluation.

During the next season, Windsor, Schmidt and Napoleon cherries were treated with the reclaimed brines and later finished into maraschinos. No consistent differences in pitting force, firmness, or reflectance were found between the fruit which had been treated with the dry mix (control) and reclaimed Napoleon and Windsor brines (Table 3). However, some differences in appearance among the maraschino cherries were noted by visual examination of the product. Napoleon and Schmidt cherries which had been brined in Napoleon brine fractions IV and V, respectively, appeared darker than the other samples even though reflectance data for these samples showed no difference.

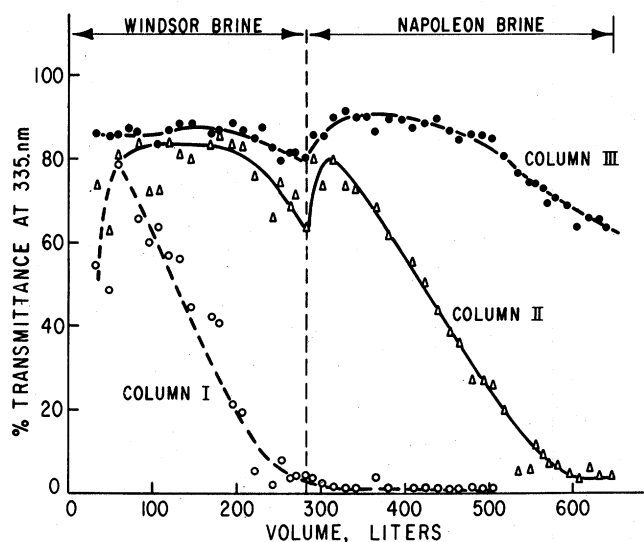


Fig. 2—Spectrophotometric evaluation of effluents from individual activated carbon columns in brine reclamation system, using per cent transmittance at 335 nm (brine pH = 3.6).

These fractions (IV and V) correspond on Figure 3 to the region beyond the breakthrough of components absorbing at 540 nm (at pH 8.0). Brines in this region show a gradual decline in transmittance at 335 nm but little absorbance at 510 nm (pH 1.0) which would be indicative of anthocyanins.

One can estimate the capacity of the system from these data, assuming that the volume of treated brine at which the breakthrough occurs is the endpoint. This volume, 500 liters, represents 282 liters of Windsor brine and an additional 220 liters of less pigmented Napoleon brine which were treated by the system. The endpoint would be 375 liters for Windsor brine alone or 887 liters for Napoleon brine, assuming that the pigment concentrations of these brines are proportional to their absorbance at 540 nm (based on our observation of the linear relationship between absorbance and brine concentration). This would be equivalent to adsorptive capacities of 95.2 and 225 ml brine/g activated carbon for the Windsor and Napoleon brines, respectively.

These values are somewhat lower than the adsorptive capacity reported by Soderquist (1971) for Black Republican cherries. His estimate was based on spectrophotometric data obtained at 335 nm rather than at 540 nm. The latter wavelength provides greater sensitivity for the detection of column saturation and the breakthrough of components which may interfere with the bleaching action of the reclaimed brine (Beavers et al., 1970).

Iron contamination

Following sand filtration and activated carbon treatment with newly prepared columns, some brine samples were observed to turn yellow slowly on exposure to air. These samples were analyzed for iron, a known cause of color problems with brined cherries (Beavers et al., 1970), and were found to contain 61.4–76.2 ppm Fe (Table 4). Analyses of the charcoal, sand, and other brine samples indicated that the discolored brine contained an abnormally high iron content before treatment. This iron contamination resulted in part from corrosion of the steel drums in which the brine was stored; the corrosion was due to the failure of protective coatings. Brine samples taken from drums generally contained more iron than did brine stored in bulk in wooden tanks or plastic lined pits. Additional iron sufficient to induce discoloration was leached

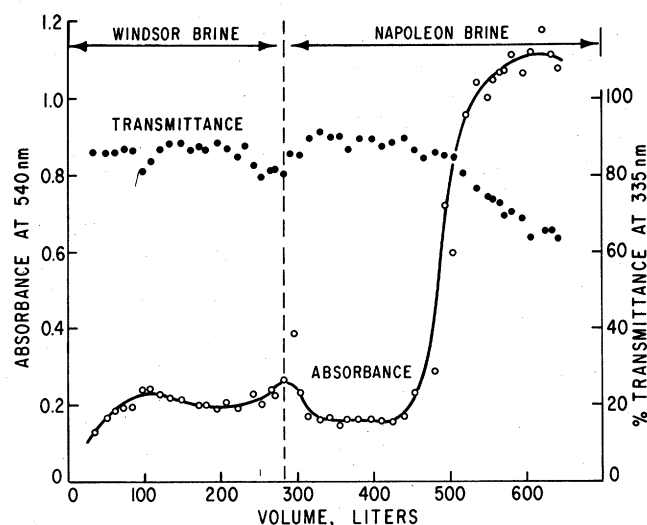


Fig. 3—Spectrophotometric evaluation of reclaimed brine following activated carbon treatment, using absorbance at 540 nm (brine pH = 8.0) and per cent transmittance at 335 nm (brine pH = 3.6).

out of the sand during brine treatment. The problem could be corrected by acidwashing the treatment columns with 1N HCl and then water washing until the eluate was acid-free.

Subsequent studies demonstrated that noticeable discoloration occurred when the iron concentration in brine exceeded 30–40 ppm. Color formation occurred immediately with ferric ion and slowly with ferrous ion, the latter reaction taking place at the air/brine interface. Presumably ferric ion reacts with a brine constituent, possibly a polyphenol, to form a colored complex. Kitson and Strachan (1955) attributed discoloration in maraschino cherries to the reaction of iron with tannins. Cohee and Nelson (1951) reported that discoloration occurred in cherries containing as little as 25 ppm iron.

During storage following treatment, a yellow discoloration was noted in Windsor brine samples containing 15 and 30 ppm Fe; the color being more intense at the higher iron concentration. This color gradually faded and was no longer evident after 21 wk at room temperature. Discoloration did not occur in the same brine which had been adjusted to 15,000 ppm SO₂ prior to storage. This transient effect may be due to the formation and subsequent degradation of a ferric ion-polyphenol complex.

Maraschino cherries made from fruit bleached with the high and low iron Windsor brines prior to finishing showed no difference in appearance or composition. Presumably, the concentration of iron in the fruit processed with high iron brine was lowered by dilution below a level which would produce noticeable discoloration.

Storage stability of reclaimed brine

Used brines are generated by cherry processors throughout the year. If the brine were to be reused, it would have to be stored for an extended period of time until the next cherry harvest. Reclamation could be carried out before or after storage. Preliminary data, obtained with used dry mix brines from the 1973 Michigan pack, indicated that untreated and reclaimed brines were similar in stability during 24 wk storage at room temperature. In this study, samples of reclaimed brine were stored for 21 wk at room temperature (Table 5). During storage, SO₂ concentrations in the brines decreased; losses were greater in full strength brines than in the reclaimed brines which had not been made up to full strength. The latter samples decreased in pH. These changes were due to the loss of SO₂ and to the oxidation of bisulfite to bisulfate, the latter reaction resulting in a pH change because bisulfate is a stronger acid. Changes in SO₂ and pH were similar with Napoleon brines containing low (Fraction I) and high (Fraction V) pigment levels and with Windsor brines containing low (12 ppm) and high (32 ppm) iron levels.

In order to minimize the loss of SO₂ from reclaimed brine, it would be best to make it up to full strength immediately prior to the start of the cherry harvest. Payne et al. (1969) recommended that new brine be used within 48 hr of preparation. However, in designing a reclamation system, this factor must be weighed along with other considerations such as the size of the reclamation columns and makeup unit and storage capacity.

Stability of water-packed cherries

Some briners are now repacking brined cherries in water for shipment to finishers. Similar procedures have been developed for the salt-free storage of olives (Martin and Robe, 1973). This practice, which shifts the burden of disposal from the finisher to the briner, could result in stability problems during storage. The data summarized in Table 6 were obtained from cherries showing little or no visible signs of change. In these samples, the pH and SO₂ content declined during storage, a probable consequence of the oxidation of bisulfite to bisulfate. Changes were greater in water-packed samples than in the brine-packed controls. Other samples showed even larger decreases in pH and SO₂, accompanied by color changes (below

200–400 ppm SO₂) and calcium sulfate precipitation. Such changes were greater in containers which were loosely capped, underfilled, or had a relatively high surface to volume ratio. Samples having low SO₂ concentrations were similar to higher SO₂ replicates in iron concentration (< 10 ppm) and showed no evidence of microbiological activity.

Maraschino cherries were made from the water- and brine-packed fruit after 1 yr of storage at room temperature. The water-packed fruit was less firm than the brine-packed fruit but similar in color (Table 7).

These results indicate that long-term storage of brined

Table 2—Transmittance and absorbance of reclaimed brines

Brine	Volume reclaimed (liters)	% Transmittance		
		335 nm (pH 3.6)	540 nm (pH 8.0)	Absorbance 510 nm (pH 1.0)
Windsor	Untreated	0.5	0.52 ^a	0.76 ^a
Windsor	108	84	0.24	—
Windsor	180	87	0.20	—
Windsor	282	80	0.28	<0.03
Napoleon	Untreated	0.9	0.22 ^a	0.22 ^a
Napoleon I	424	87–91	0.14–0.17	0.000
Napoleon II	480	84–87	0.23–0.29	—
Napoleon III	504	85	0.60–0.72	0.005
Napoleon IV	565	73–81	0.96–1.07	0.01
Napoleon V	641	63–66	1.08–1.18	0.01–0.02

^a 1/10 dilution

Table 3—Effect of reclaimed brine on quality of maraschino cherries—1975 season

Cherry variety	Brine	Shear press ^a		Reflectance		
		Pitting force	Firmness	Rd	a	b
Windsor	Control	7.4	7.6	6.0	46.8	18.6
	Napoleon I	7.8	7.7	5.8	44.9	18.2
	Windsor	9.5	6.5	5.9	43.9	17.7
Schmidt	Control	10.2	9.4	6.4	51.0	19.4
	Napoleon I	10.5	9.4	7.1	54.9	20.3
	Windsor	10.6	8.9	6.3	48.5	18.5
Napoleon	Control	9.7	9.4	5.8	44.5	18.2
	Napoleon II	9.2	8.5	6.3	48.8	18.9
	Windsor	9.5	8.9	5.6	43.7	18.4

^a Area under curve/g sample; pitting force for brined cherries and firmness for maraschino cherries

Table 4—Iron-induced discoloration of reclaimed brine

Sample	Iron (ppm)	Brine color
Reclaimed brine, drum 1	61.4–76.2	Yellow
Untreated brine, drum 1	36.0–39.3	Normal
Untreated brine, other drums	12.8–23.0	Normal
Untreated brine, stored in bulk (37 samples)	0.6–8.9	—
Sand	22.5	—
Activated carbon	3.0–3.5	—
Reclaimed brine, drum 1 after system acid-washed	30.8–32.5	Normal

Table 5—Storage stability of reclaimed brines

Brine ^a	pH					SO ₂				
	Storage time (wk) ^b					Storage time (wk) ^b				
	0	5–6	12	16–17	19–21	0	5–6	12	16–17	19–21
Nap I	3.64	3.52	3.46	3.52	3.17	4540	4470	4190	4190	4140
Nap V	3.61	3.60	3.62	3.29	—	4440	4340	4340	4240	—
W-low Fe	3.74	—	3.38	3.28	3.30	4100	—	3450	3150	3100
W-high Fe	3.77	—	3.42	3.28	3.30	3940	—	3360	3100	2880
Nap I - FS	2.80	2.80	2.90	2.80	2.80	16400	14300	14300	14400	14400
W-low Fe - FS	2.80	2.80	2.76	2.91	2.80	15800	14300	14000	14000	14100
W-high Fe - FS	2.80	2.77	2.70	2.92	2.80	16000	14200	13600	13700	13500

^a FS = Full strength^b Room temperature

Table 6—Stability of brined cherries packed in water and brine

Cherry variety	Storage (mo)	Water-pack			Brine-pack ^a		
		pH	SO ₂ (ppm)	Color ^b	pH	SO ₂ (ppm)	Color ^b
Napoleon	1	3.4	1490	Y	3.4	2380	Y
	6	2.9	1350	Y	3.3	2810	Y
	9	3.1	950	Y	3.1	2260	Y
Windsor	1	3.6	1680	Y	3.6	3200	Y
	6	3.1	560	Y-O	3.6	2900	Y
	9	2.8	160	O	3.4	2510	Y

^a Dry mix brine^b Y = Yellow; O = OrangeTable 7—Quality of maraschino cherries made from water- and brine-packed fruit^a

Cherry	Brine	Water-pack				Brine-pack			
		Firm-ness ^b (AUC/g)	Color			Firm-ness ^b (AUC/g)	Color		
			Rd	a	b		Rd	a	b
Napoleon	SO ₂	4.9	4.2	31.5	7.6	5.7	4.4	32.6	8.4
	Dry mix	4.3	4.2	32.3	7.9	6.3	4.3	33.6	8.0
Windsor	SO ₂	6.3	4.2	30.3	7.4	8.6	—	—	—
	Dry mix	4.9	4.0	30.0	7.0	5.4	—	—	—

^a Stored for 1 yr at room temperature before finishing^b AUC/g = area under curve/sample weight

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cherries in water may result in oxidation and softening which could affect product quality. Water packing can be used to alleviate the finisher's waste disposal problems and to improve the logistics of brine recycling. However, this practice will reduce the storage life of the product and may necessitate the use of corrosion-resistant airtight containers.

Future research needs

An engineering study and cost analysis are required to establish the economic feasibility of brine reclamation. These analyses must consider the logistics of brining and finishing, disposal costs, storage losses, the strategy of water packing brined cherries, and the cost of the unit operations of brine treatment. Such a program is now in progress at Michigan State University.

CONCLUSION

RECYCLING of spent cherry processing brine will reduce waste disposal requirements without adversely affecting product quality.

Chemicals, equipment, and storage containers must not contribute iron to the treated brine to avoid discoloration.

Water packing of brined cherries is not compatible with longterm storage unless precautions are taken to prevent oxidation and softening.